EXPERIMENTAL STUDIES ON FRICTION STIR PROCESSING (FSP) OF COMPOSITES

A

Synopsis

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Submitted by

Pratap Singh

Under the supervision of

Prof. Rahul Swarup Sharma
(Co-Supervisor)
Department of Mechanical Engineering
Faculty of Engineering

Dr. Ankit Sahai
(Supervisor)
Department of Mechanical Engineering
Faculty of Engineering

Department of Mechanical Engineering, Faculty of Engineering

Dayalbagh Educational Institute (Deemed University)

Dayalbagh, Agra (UP) – 282005

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1.1 Friction Stir Processing

Friction stir processing (FSP) is a new severe plastic deformation technology that can be utilized for surface modification, fabrication of surface composites, modification and control of microstructure, enhancement of mechanical and thermal properties, etc. It is mostly used with Al based alloys for improving mechanical properties such as hardness, tensile strength, etc. Further this technique can be used for making surface composites and can improve the surface properties such as abrasion resistance, hardness, strength, ductility, corrosion resistance, fatigue life and formability without affecting the bulk properties of the materials.

FSP in its simplest form consists of a non-consumable rotating tool, which is plunged into a work piece and then moved in the direction of interest. The schematic illustration of FSP is shown in Fig. 1(a) and 1(b).

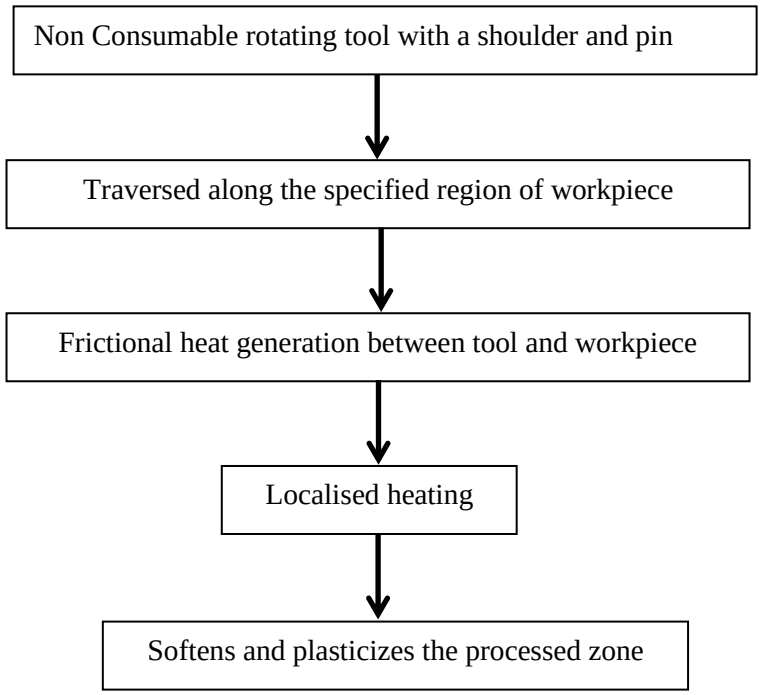
Non Consumable rotating tool with a shoulder and pin
Traversed along the specified region of workpiece
Frictional heat generation between tool and workpiece
Localised heating
Softens and plasticizes the processed zone

Fig. 1 (a) Schematic steps of FSP technique

The heat is generated mainly by the friction of the rotating shoulder with the work-piece, while the rotating probe or pin stirs the heated material. The heated material softens and flows around the rotating pin. It then fills the cavity at the rear of the tool [1]. The material that flows around the tool is subjected to severe plastic deformation and thermal exposure, which leads to a significant

refinement of microstructure in the processed zone. Stir zone **(**SZ) refers to the zone stirred by the tool probe [2].

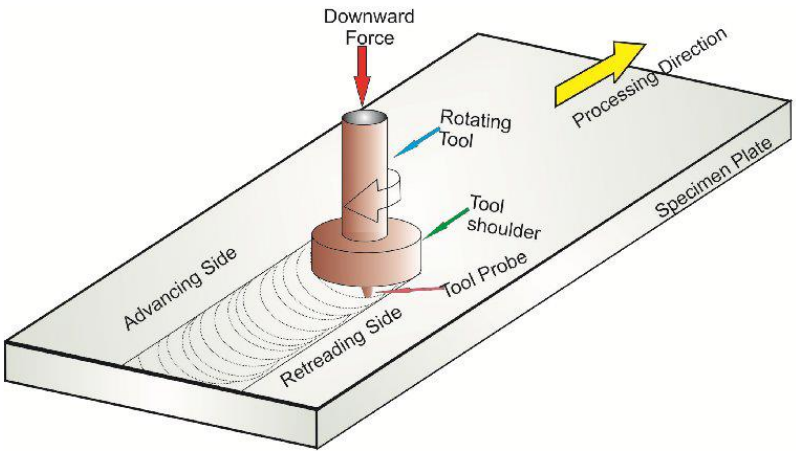

Fig. 1 (b) Schematic illustration of FSP technique

1.2 Metal Matrix Composites (MMCs)

Metal matrix composites is a composite material with a combination of at least two components, one of the base metal (usually an alloy) and other is of hard ceramic particles. Fabrication of MMCs are usually done by dispersing a reinforcement material into a metal matrix via various techniques. It includes powder metallurgy, laser technique, electron beam irradiation, spray deposition, casting, mechanical alloying, etc. All the above techniques of composite fabrication are done in liquid phase at high temperature.

FSP is a technique conducted below melting point and offers an excellent choice for surface modification as well as bulk composites of metal alloys. The first journal discussing FSP was published in 1999 [3], and thereafter number of papers, patents and even textbooks are available covering this topic. The initial applications of FSP were reported on wrought aluminium alloys as a grain refinement method to provide superplasticity [3], and as a mechanical consolidation method to homogenise microstructures of castings [4], powder metallurgy materials [5,6], and composites [7], which have been reviewed in 2008 [8].

FSP have the ability to fabricate MMCs in linear bands along the surface of plate by arbitrary combinations of particles and alloys [9]. This can be happened by making either grooves or blind holes drilled into the surface which are later filled by the reinforcement particles (RPs). Nanosized RPs enhanced Orowan looping strengthening mechanism and also refining grain structures by

suppressing grain growth via grain boundary pinning [10]. However, a precaution should be taken in selecting an alloy, since peak-aged alloys may experience MMC softening in the stir zone which offsets the benefits from incorporating other particles [11]. Fabrication of composites using nanoparticles offer the greatest strengthening benefit, seeing the Orowan looping and grain boundary pinning mechanisms since it is possible to scatter nano-scale particles which have an illogical interface with the matrix, whereas earlier precipitation-aged aluminum alloys contain nano-scale particles with logical or semi-logical interfaces [12-14].

The next section covers the detail literature of process variables incorporated in friction stir processing of composites.

2.1 EFFECT OF PROCESS VARIABLES

FSP process variable can be broadly categorized into three categories. They are:

i. Machine variables
ii. Tool design variables
iii. Material properties

Fig. 2 depicts various process variables involved in FSP. The mechanical and thermal properties of base materials are decisive in selecting process variables.

2.1.1 Machine Variables

Machine variables are those parameters which can be altered by changing the machine settings such as tool rotational speed, tool traverse speed, tool tilt angle, tool and plunge depth, axial force, etc. First two parameters are major machine variables. The amount of heat generation and the rate of material movement are affected by altering these parameters. It is clear from literature that combination of higher rotational speeds and lower travel speed increases heat input, material flow and consequently improves particle distribution [15-17].

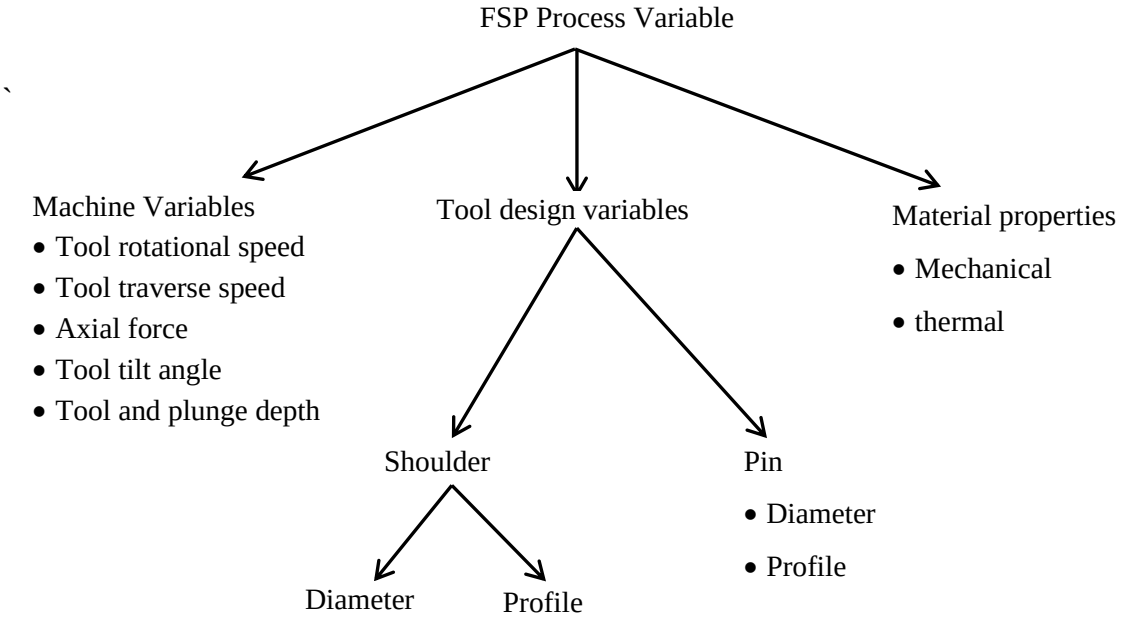

Fig. 2. Classification of FSP process variables

Arbegast and Hartley, 1998 proposed an empirical relationship between rotational speed (ω) and traverse speed (v) with the maximum temperature (Tmax) in SZ [18].

$$\frac{\text{Tmax}}{Tm} = K\left(\frac{\omega^2}{v\ X\ 10^4}\right)^{\alpha}$$

where, Tm is the melting point temperature in ºC, K is the constant and lies between 0.65 to 0.75, and the exponent α ranges from 0.04 to 0.06.

Fu et al., 2013 [19] demonstrated that the relationship between peak temperature (Tmax) and heat index value is given by

$$Q = \frac{4}{3}\pi^2 \frac{\alpha\mu P\omega R^3}{v}$$

where, α is the heat input efficiency, μ denotes friction coefficient, P is the pressure (in Pa) and R is the tool radius (in m). From the above relation, it is clear that the heat input directly relates with the rotational speed and inversely relates to traverse speed.

Chen and Kovacevic [20] considered both shoulder as well as probe radius and proposed that rate of heat generation over the entire interface is given by

$$Q = \frac{2\pi\omega\mu P({R_0}^3 - {r_0}^3)}{3}$$

where, μ is coefficient of friction, ω is angular velocity, P is axial pressure, R_o is the tool shoulder radius and r_0 is the tool pin radius.

Other parameters include axial force, tool tilt angle and tool and plunge depth. Axial force affects the particle distribution. At low axial force the particle distribution is homogenous and at high force particles are ejected out. [11,21]. So, an adequate axial depth is required for uniform distribution of particle.

The tool tilt angle and tool plunge depth are interrelated and effect the formation of shear zone (SZ). [22]. If plunge depth remains constant and the tool tilt angle increases, lesser surface of the tool shoulder touches the workpiece results lesser heat generation. Asadi et al. [23] suggested the optimum plunge depth required for tilt angles 2.5º, 3º, and 3.5º were 0.22, 0.30, and 0.40 mm, respectively.

2.1.2 Tool Design Variables

The tool geometry plays an important role in friction stir processing. It affects the heat generation, reinforcement distribution and physical and microstructural properties of material [24]. The friction between rotating tool and workpiece causes the heat generation. Heat generation is also occurred during plastic deformation of material [25].

Shoulder and pin design are the main aspects of tool design. Shoulder design involves tool diameter and angle of end surface of shoulder. Tool pin consists of pin diameter, pin length, and shape of pin. All the parameters of tool design are important and play a vital role in designing of tool of friction stir processing.

It is clear from the equation below; the heat input is a function of coefficient of friction between tool and workpiece, applied pressure/force, rotational speed and shoulder radius to the third power. [26, 27]. Therefore, the heat input in FSP is strongly dependent on the shoulder size. Also, the axial force is a function of the shoulder radius [28, 29].

$$q_0 = \frac{4}{3}\pi^2 \mu P \omega R^3$$

where, q_0 is the net power (in W), μ is the effective friction coefficient between the tool and the workpiece, P is the pressure (in MPa), ω is the rotation speed (in rev/ min), and R is shoulder radius (in mm).

The optimum tool shoulder diameter follows the straight line equation with workpiece thickness having a slope of 2.2 and intercept value of 7.3 mm [30]. For optimal weld properties,

shoulder to pin diameter (D/d) ratio of 3 were generally used. [24,31]. In the study of Vijayavel et al. [32], ratio of 3 exhibited better microstructural and mechanical properties.

In addition to shoulder diameter the end surface shape also affects the material flow. Shoulder end can either have flatness, concavity and convexity (Fig. 3). Flat shoulder end surface causes the excessive flash during FSP. The contact of workpiece at any location can be attained during FSP with convex end surface shoulder. However, in concave end shoulder, the tool tends to lift upwards because during FSP at a high travel speed, the stirred material is accommodated in the cavity under the shoulder. The end surface may also be different profiles such as scrolls, ridges, spiral etc.

The design of the probe is also important. The movement of material and distribution of reinforcement particles are mostly affected by the probe profile. The end shape of the probe is either flat or domed (Fig. 4). The flat bottom probe is of common form and requires high forge force during plunging. While, in domed end shape probe the pressure is reduced and increases the tool life. The tapered probe is also one of the alternatives. Larger contact area of probe with the workpiece during FSP with tapered probe increases frictional heat results higher plastic deformation.

The probe outer surfaces can have different shapes and features including threads, flats and flutes (Fig. 4). The particle distribution was found more homogeneous with a square pin as compared to circular and triangular pins [33]. The square pin profiles result in more grain refinement as compared to non-threaded circular pins. [34]. Threaded pin possesses higher homogeneity as compared to cylindrical and square pin [35, 36].

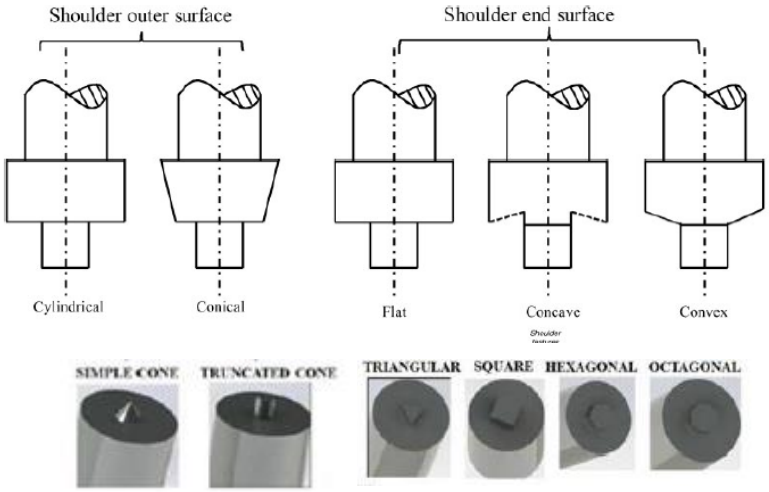

Fig.3 Shoulder shapes

2.1.3 Material properties

The properties of material contribute a key role in selection of process parameters. Shear effects affected by material's mechanical properties and amount of heat generation could be affected by material's thermal conductivity. In past decades, various authors conducted experiments with different process parameters used for FSP of different metal alloys.

2.2 FABRICATION OF SURFACE COMPOSITES (SCs) VIA FSP

FSP technique involves exterior layer modification, which improved surface properties while retaining the bulk properties unchanged. During initiative years, FSP was employed in development of SCs of light metal alloys like aluminum. But, nowadays, it has gained a shining role in the field of SC fabrication of various nonferrous alloys like aluminum, magnesium, copper, and even ferrous metals like steel.

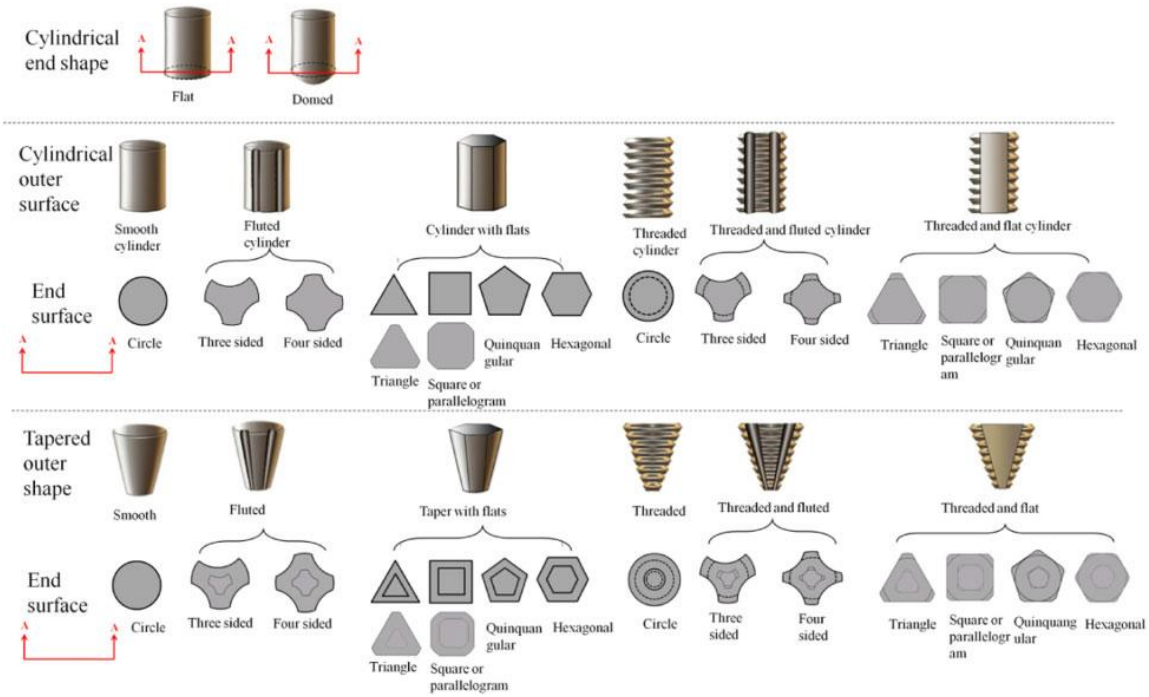

Fig. 4 Different probes shapes used during FSP/W. Source: Zhang et al., 2012 [37]

In many industries, light metals such as non-ferrous alloys are rarely used because of their low weight density, stiffness and strength. However, if the strength of these light metals will improve, they are more advantageous to industries like aircraft, marine, automobile, etc. Reinforcement of such metals is required to improve the metallurgical, mechanical and thermal properties.

According to Rathee et al, (2017) [38], there is potential of lot of improvement in the direction of single pass FSP. Suitable design of a tooling system or process variants in FSP needs further investigation which can address aforementioned research issues thereby make FSP more effective technique by saving time and energy. Another interesting research aspect is difficulty encountered in uniform dispersion of nano-sized RPs when compared to micro-sized RPs [39]. Tool wear and its variation with RPs type as well as process parameters during SC fabrication using FSP has not been investigated till now. Despite a multitude of relevant research, a few aspects like material flow aspects have not been fully understood.

Sharma et al, 2015 [9] found the research gap in research on surface composites via FSP route such as fabrication of defect-free composites, tailoring microstructures, development of durable and cost effective tools, and understanding on the strengthening mechanisms.

2.2.1 Reinforcement variables

The effect of reinforcement particles in surface composites during FSP plays a vital role. There are mainly three types of reinforcement variables. They are:

i. Type of RPs
ii. Size and fractional volume of RPs
iii. Strategy of reinforcement used

Type of reinforcement particles

In literature various types of reinforcement particles were reported like silicon carbide (SiC) [40], boron carbide (B_4C) [41], titanium carbide (TiC) [42], aluminum oxide (Al_2O_3) [43], silicon oxide (SiO2) [44], titanium oxide (TiO2), carbon particles like graphite [45], etc. The selection of reinforcement is done depending upon the application of end-use products. In addition to mono composites, several authors produced hybrid composites using more than one type of RPs at different hybrid ratios [46,47].

Size and fractional volume of RPs

Generally, RPs used in SC fabrication are of micrometre or nanometre range. The size and fractional volume of RPs has great impact on microstructural as well as mechanical properties of fabricated SCs [48−50]. It is well known that the small sizes of RPs result in better properties.

Strategy of reinforcement

Reinforcement strategy can be broadly classified under three heads as shown in Fig. 5.

Direct pasting technique of RPs was first utilized by Mishra et al [51]. In this technique RPs mixed with some volatile solvent and directly pasted on the base metal [52, 53] or by spraying of RPs by a suitable method like plasma spray [54], high velocity oxy fuel spray, etc. [55, 56]. A major drawback of this technique was to spatter the RPs.

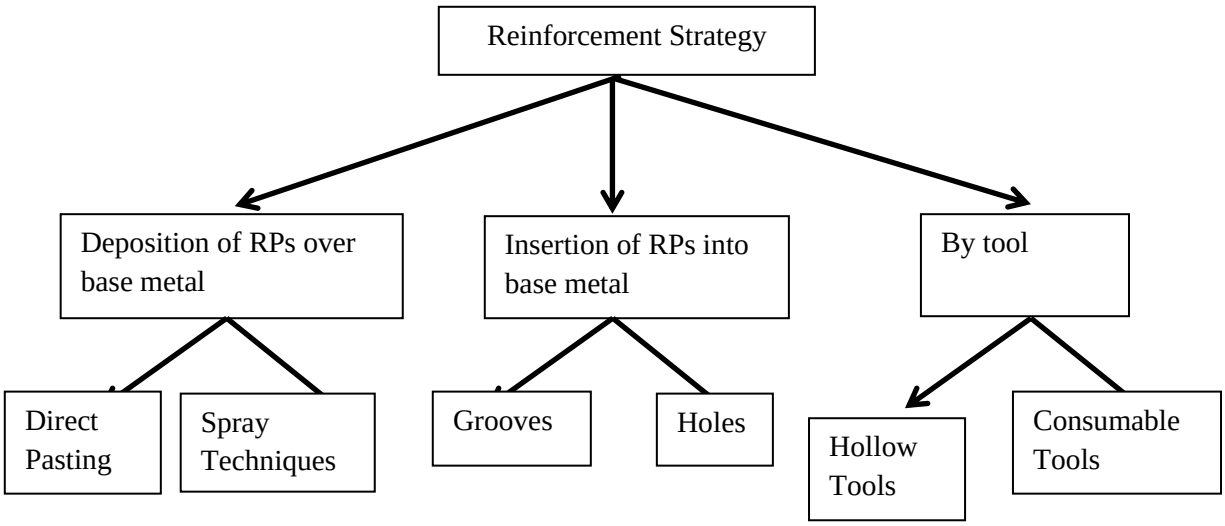

Fig.5. Classification of reinforcement strategies

To overcome this problem another strategies used which are explained ahead. Zahmatkesh and Enayati [55] used this technique to improve the hardness of composites material AA2024/Al_2SO_4 of 230 Hv as compared to hardness of 90 Hv of base metal of AA2024. Mazaharii et al. [56] utilized high velocity oxy-fuel (HVOF) spray technique to A356/Al_2O_3 SCs and found satisfactory results with negligible flaws. A typical arrangement of this technique is shown in Fig. 6.

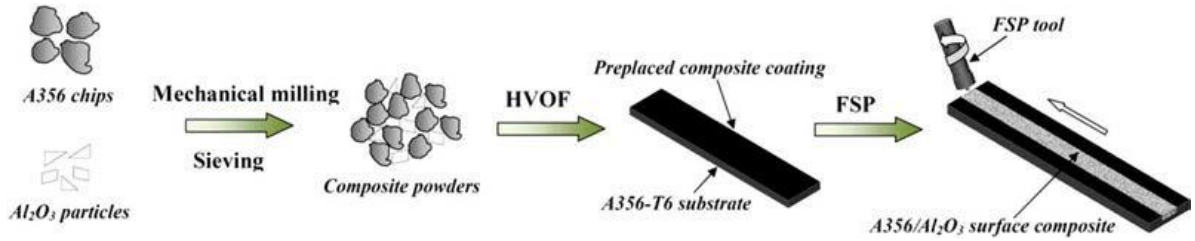

Fig.6. Procedure used in the HVOF spraying technique. Source: Mazaheri et. Al. [56]

During the last decade, the most commonly used reinforcement technique is the groove technique [25,57,58]. The schematic illustration of SC fabrication using the groove technique is shown in Fig. 7. A probe less tool is applied for closing groove opening to avoid sputter of RPs during FSP. In some studies, the grooves of holes were covered by a thin sheet to avoid RP ejection [59]. As shown in Fig.8 (a).

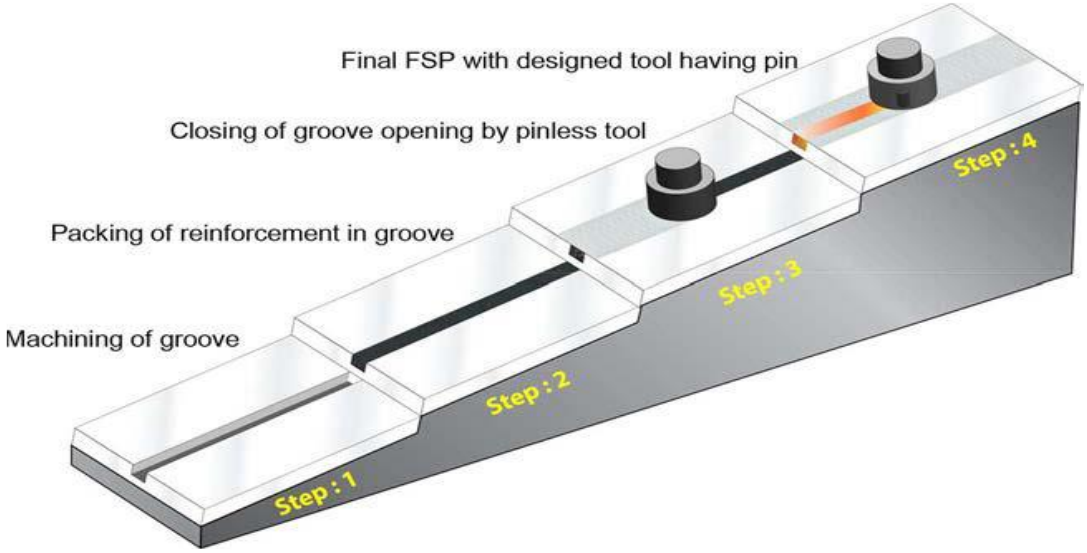
Final FSP with designed tool having pin
Closing of groove opening by pinless tool
Packing of reinforcement in groove
Machining of groove
Step : 1
Step : 2
Step : 3
Step : 4

Fig. 7 Steps in SC fabrication using the groove technique. Source: Rathee et. al. [38]

To avoid the covering another method of blind drilled holes (Fig. 8 (b)) were reported [60, 61]. The loss of RPs was restrained automatically because half part of shoulder ahead of traversing pin seals the opening of the holes. However, to reduce the machining time an alternative approach of direct friction stir processing process (DFSP) was proposed by Huang et al. [62] (Fig. 8 (c)).

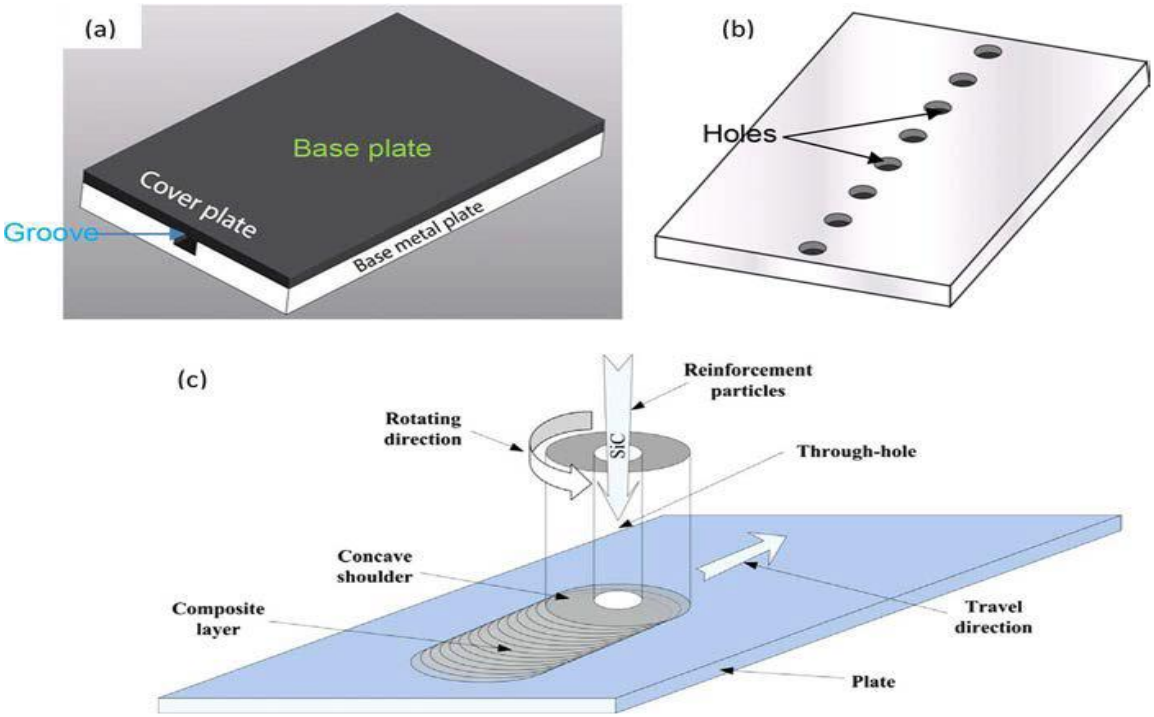

Fig.8 Schematic representation of (a) covering of grooves by thin plates, (b) blind drilled holes for RPs, (c) DFSP method. Source: Huang et al. [62].

Few gaps in research on surface composites via FSP route such as fabrication of defect-free composites, tailoring microstructures, development of durable and cost effective tools, and understanding on the strengthening mechanisms. Homogeneous dispersion of nanofillers is critical in achieving properties of nanocomposites. Therefore, optimizations of process parameters are still required. To overcome the challenge of tool wear during processing with hard ceramic particle is still required. Another major issue includes extremely time consuming processing, which involves cutting a groove or drilling holes, filling the cavities with RPs, and multiple FSP passes. In spite of a wide range of studies on composites using different particles, a clear correlation between the mechanical behaviours of composites with the particle properties remains unclear.

2.3 INTEGRATION OF FRICTION STIR PROCESSING (FSP) AND ADDITIVE MANUFACTURING (AM)

Over the last decades, FSP and AM – each technique with its own characteristics – have developed as dominant techniques for the manufacturing of light alloys and their composites. Till now, the integration of both these techniques is not being explored much. So, in this work, an attempt will be made to explore the integration of FSP with AM, to understand the correlations between processing parameters – microstructure and properties in the FSP and AM of light alloys, their composites and

polymers. Lim et al., 2021 [63] conducted FSP on 3D printed metal composites. They found significant grain refinement and improvement of ductility in the specimens of Friction Stir Processed (FSPed) 3D printed metal composites. Du et al., [64] also performed FSP on 3D printed Al alloys and observed a significant decrease in microhardness in the processed region.

The Fused Deposition Modeling (FDM) is one of the most commonly used, a layer by layer AM technique. 3D printing technology is gaining its popularity in various types of industries such as automobile, aerospace, bio-medical, food processing, civil structures etc. This technology reduces the cycle time and hence production cost [65]. The main advantage of FDM based 3D printing is to produce complex to complex object with greater ease, reasonable price and in quick time with better quality. In conventional subtractive manufacturing process, more time, cost, and energy are required. Therefore, industries are now-a-days trying to replace such conventional subtractive ways of manufacturing with Additive Manufacturing (AM) techniques. Though, AM has potential to revolutionize industries and manufacturing methods, but have some limitations and drawbacks (lower mechanical properties, limited size of object, lower production rate). In this work, Investigation of the effect of FSP on the FDM based 3D printed specimens can be carried out and analyse the mechanical properties and morphological structures for polymers.

The most popular Fused Deposition Modeling technique is used in this work for fabrication of 3D printed specimen for FSP. In this technique, a plastic filament wire is feed into a liquefier chamber as shown in Fig. 9. In 3D printing, generally used plastics are Acrylonitrile Butadiene Styrene (ABS), Polycarbonate (PC), Polylactic acid (PLA), etc. The 3D printed parts by using such raw materials have lower mechanical properties which limit their use in engineering applications. So, in order to improve the mechanical properties of the 3D printed object, reinforcement particles can be added by any reinforcement technique as discussed in this synopsis with the help of FSP. In some studies, carbon fibre reinforced polymer composites have potential to meet out acceptable mechanical properties as required for industrial used products [66,67].

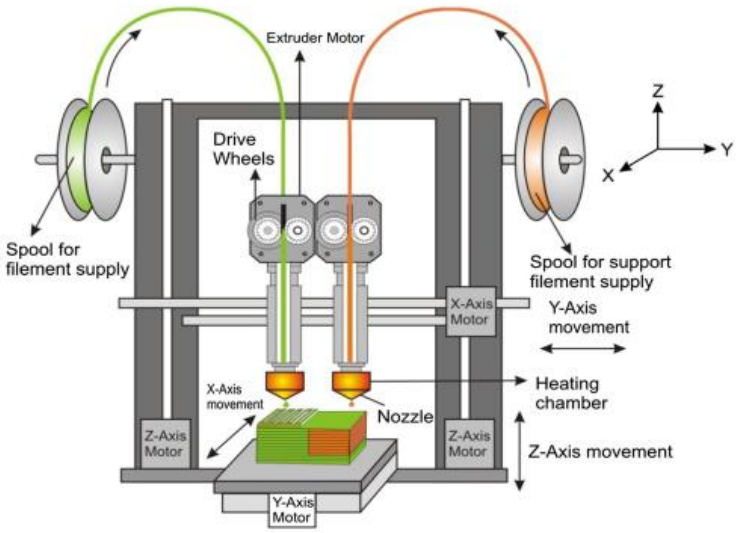

Fig. 9 Fused Deposition Modeling machine setup. Source: Yadav et al. [68].

3. OBJECTIVES

1. The proposed materials to be used in this study are aluminium alloys with different reinforcement particles like multi-walled carbon nanotubes, SiC, etc.
2. The techniques to be involved for reinforcement are as follows:
    i. Groove technique
    ii. By using thin sheet to avoid RP ejection
    iii. Blind drilled holes
3. Experimental investigation the effects of different process parameters on morphological and mechanical properties of metal matrix composites. These parameters are:
    i. Number of passes
    ii. Different pin profiles
    iii. Rotational and traverse speed
4. To investigate the integration of FSP and Additive Manufacturing (Fused Deposition Modeling) for polymer composites.
5. Friction Stir Processing of Additive Manufactured polymer composites to analyse morphological and mechanical properties.

4. METHODOLOGY AND WORK PLAN

The methodology and work plan used for achieving the aforementioned objectives is described below one by one:

Objective 1: The proposed materials to be used in this study are aluminium alloys with different reinforcement particles like multi-walled carbon nanotubes, SiC, etc.

The materials used in this work are Al 6061 and Al 7075. We mainly focus on Al 6061. The different specimens of same dimensions are prepared. Then, reinforcement particles are incorporated using different methods as discussed in objective 2. The specimens with different reinforcement particles like SiC, Al_2O_3, multi-walled carbon nanotubes (MWCNTs), etc. are prepared. Then, FSP will be done on the specimens with or without reinforcement.

Objective 2: The techniques to be involved for reinforcement are as follows:

i. **Groove technique**
ii. **By using thin sheet to avoid RP ejection**
iii. **Blind drilled holes**

In literature, a groove technique is mostly used for reinforcement. In this technique, a groove is made by conventional milling machine, then RPs is filled in that groove, then a pin-less FSP tool is traversed over the machined groove to close the opening of groove and then final FSP is performed with designed tool having pin. In some studies, the machined groove with reinforcement is covered by a thin sheet to avoid ejection of RPs. In blind drilled holes technique, the ejection of RPs is restrained automatically because half part of shoulder ahead of traversing pin seals the opening of the holes during FSP.

Objective 3: **Experimental investigation the effects of different process parameters on morphological and mechanical properties of metal matrix composites. These parameters are:**

i. Number of passes
ii. Different pin profiles
iii. Rotational and traverse speed

The effect of these parameters on mechanical properties will be investigated. We will focus on tensile strength, microhardness, impact, etc. The fractography analysis will be done for tensile tested specimen to understand the fracture mode during deformation.

Objective 4: To investigate the integration of FSP and Additive Manufacturing (Fused Deposition Modeling) for polymer composites.

The Fused Deposition Modeling (FDM) is most commonly used material extrusion technique. FSP can be carried out on the specimens developed by FDM. The plastic materials to be used in this work are Acrylonitrile Butadiene Styrene (ABS), Polycarbonate (PC), Polylactic acid (PLA), etc. In the FDM technique, a plastic filament wire is feeds into a liquefier chamber. Firstly, we prepare a specimen design using any designing software (Solidworks, AutoCad, Catia), this CAD file is converted into .STL format which is easily understands by printer. Then, a Cura software is used where the slicing of the designed object is done, G-code is prepared which is fed into the printer through SD Card or pen drive and optimum parameters are decided for the specimen to be printed. The FDM based 3D printer reads this G-Code and the nozzle of the printer moves according to the coordinates of the model to be printed. Thus, the printer prints the required specimen. A comparative study will be done on the additive manufactured specimens with and without FSP.

Objective 5: Friction Stir Processing of Additive Manufactured polymer composites to analyse morphological and mechanical properties.

The specimens developed by additive manufacturing are lighter in weight and having lower strength. In this work, an attempt will be made to improve the strength of additive manufactured polymer by Friction Stir Processing. The mechanical properties for example tensile strength, compressive strength, microhardness, etc. and morphological analysis will be done on the different specimens prepared by FDM. By altering different process parameters (rotational and traverse speed, number of passes, different pin profiles), different specimens will be prepared and the mechanical properties will be investigated. Surface profiling and microstructure analysis will also be performed on Friction Stir Processed 3D printed samples.

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